



Docket No.: 20523 US (C038435/0120240)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:

Chyi-Cheng CHEN and Bruno LEUENBERGER)

Examiner: L. Channavajjala

Serial No.: 09/726,880)

Art Unit: 1615

Filed: November 30, 2000)

For: **A VITAMIN POWDER COMPOSITION
AND METHOD OF MAKING**)

DECLARATION OF MR. HERMANN STEIN UNDER 37 C.F.R. §1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Hermann Stein, a citizen and resident of Switzerland, hereby declare as follows:

1. From April 1958 until September 1961 I studied chemical laboratory assistant at Farbwerke Hoechst AG, Frankfurt/Main Hoechst, Germany. From April 1963 until March 1966 I studied chemical engineering at Paul-Ehrlich-Schule, Frankfurt/Main, Germany.

2. Employment history:

- April 1958 - March 1963 chemical laboratory assistant at Farbwerke Hoechst AG
- April 1963 – March 1968 chemical laboratory assistant and chemical engineer at Battelle-Institut e.V., Frankfurt/Main, Germany. Development of new high temperature polymers and new resins for cosmetic applications.

- April 1968 – November 1982 at F. Hoffmann La Roche AG, Basle Switzerland, development of new cosmetic products, starting as head of development laboratory and finally chief of cosmetic development department.
- December 1982 – January 1984 at Vick International Paris, development of new cosmetic products and know how transfer cosmetic department Hoffmann La Roche to Vick International.
- February 1984 – January 2002 at F. Hoffmann La Roche AG, Basle, Switzerland and later on Roche Vitamins AG, Basle Switzerland: Development of new vitamin product formulations, improvement of existing vitamin product formulations and development of new production processes for vitamin product formulations. Position: Head of development laboratory and mini plant, process engineer for pilot plant production trials.
- Since March 2002, I have been a technical consultant for Roche Vitamins AG and DSM.

3. The present application discloses and claims powder compositions containing at least one fat-soluble vitamin dispersed in a matrix consisting of an emulsion-forming composition selected from the group consisting of a natural polysaccharide gum, a mixture of polysaccharide gums, a protein, a mixture of proteins, and mixtures thereof, wherein the fat-soluble vitamin is present in the powder composition in the form of solid droplets having an average diameter of about 80 to about 120 nanometers (nm).

4. I am aware that a Final Office Action issued in the above-identified application on February 23, 2004. I understand that claims 1 and 3-15 were rejected under 35 USC § 103 over Stein *et al.*, EP 0 937 412 ("Stein") or Stein in view of Ford *et al.*, 5,607,707 ("Ford"). (Paper No. 02122004 at 2.) I further understand that claim 17 was rejected under 35 USC § 103 over Stein alone or Tritsch *et al.*, EP 0 841 010 ("Tritsch '010") alone or in combination with Ford in view of Finnan *et al.*, U.S. Patent No. 4,830,859 ("Finnan"). (Paper No. 02122004 at 5.)

5. I am a co-inventor of the subject matter described and claimed in the Stein document relied upon by the Examiner in rejecting the claims of the above-captioned application. Stein is entitled "PREPARATION OF A FINELY DIVIDED PULVEROUS CAROTENOID PREPARATION" and discloses "a continuous process for converting carotenoids, retinoids or natural colourants into finely divided pulverous forms which are particular[ly] required for colouring foodstuff[s] and animal feeds." (Col. 1, paragraph 0001.) In particular, Stein discloses a five step process:

- a) forming a suspension of the active ingredient in a water-immiscible organic solvent optionally containing an antioxidant and/or an oil,
- b) feeding the suspension of step a) to a heat exchanger and heating said suspension to 100-250°C, whereby the residence time in the heat exchanger is less than 5 sec,
- c) rapidly mixing the solution of step b) at a temperature in the range of 20-100°C with an aqueous solution of a swellable colloid optionally containing a stabilizer,
- d) removing the organic solvent and
- e) converting the dispersion of step d) into a pulverous preparation. (Abstract and col. 1-2, paragraph 0008.)

6. During the research that lead to the invention disclosed in Stein, my co-inventors and I, using the knowledge available at the time, attempted to produce the smallest

possible particle size. In particular, Example 5 was included with a view toward optimizing the process by producing the smallest possible particle size. Example 5 is reproduced below:

Solvent: methylene chloride, direct heat transfer (steam).

9.25 kg Fish Gelatin, 18.5 kg of sugar and 2.5 kg of Ascorbyl palmitate were dissolved in 30.25 kg of water in Kettle 1. The pH-value of this matrix was adjusted with NaOH (20%) to 7.2 - 7.6.

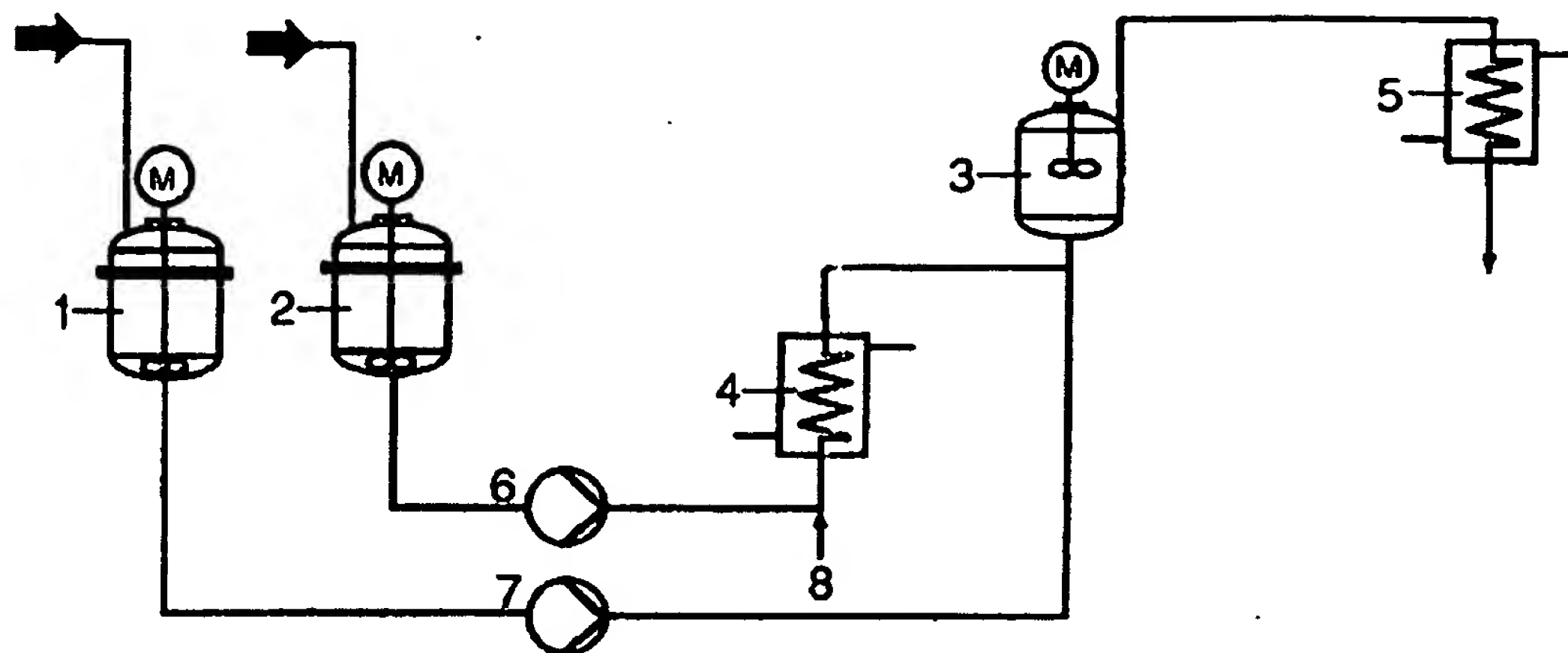
6.0 kg of β -Carotene, 0.75 kg of dl- α -tocopherol and 3.0 kg of corn oil were dispersed in 30.0 kg of methylene chloride in Kettle 2.

The β -carotene suspension was fed by pump 6 with a flow rate of 20 kg/h to the heat exchanger 4 where it was mixed with steam to reach an outlet temperature of 145°C. The residence time in the heat exchanger 4 was 1.3 sec. The matrix was pumped by pump 7 with a flow rate of 30.4 kg/h to the Kettle 3 where the solved β -carotene was mixed with the matrix and emulsified in it. The emulsion was cooled down to 35°C in heat exchanger 5.

Methylene chloride was removed from the emulsion by using a vertical evaporator. The resulting emulsion showed a particle size of the inner phase of 196 nm and was spray dried.

The final product has a β -carotene content of 9.9%, E1/1: 1120, λ_{\max} : 440-460 nm. The powder was well soluble in water, the solution has a very intensive yellow color. (Col. 6, paragraph 0054-0058.)

Figure 1 is reproduced for clarity:



7. As Example 5 shows, at that time, at best we could produce particles sizes of about 196 nm. Based on my unique knowledge of the methods and compositions of Stein, and my long experience in the area of the production carbohydrate matrices, it is my opinion that one of skill in the art at the time of the above-captioned invention familiar with the disclosure of Stein could not have produced particles of the presently claimed size.

8. Moreover, it is also my opinion that one could not have predicted that the process of the present invention would produce significantly smaller particle sizes than the methods of Stein.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of

Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: March 16, 2005

Hermann Stein
Hermann Stein